1 Purpose

This procedure is used to remove a volume of liquid water from the bubble chamber and replace it with 29 kg of CF3I .

2 Prerequisites

It is assumed that the system is at room temperature and in the “Compressed” state valve position.

It is assumed that the hydraulic system has less than 0.4 liter of accumulated air accomplished by normal purging/”burping” process. The 0.8” orifice is installed in the fast recompression line.

The bubble chamber is currently filled with de-gassed, purified water obtained from the FNAL A0 cleanroom.

3 Introductory notes

The differential pressure across the bubble chamber and quartz jar must be limited to 15 psid in either direction. No objectionable differential pressure will be developed as long as the stop plate is within its free travel range. The diaphragm tank has a normal weight range on WT-9 of 53.5 lbs to 61 lbs to provide a volume change of 3.3 liters of glycol.

CAUTION: CF3I is an irritant to the eyes and skin. It also can cause cardiac sensitization at concentrations above 0.4% for exposures exceeding 1 minute. If CF3I is accidently released into the environment, then calmly close the nearest valve to stop the release. A two person rule is in effect during active filling steps (valves being opened/closed, connections being made or un-made, etc.) in this procedure. Confined space procedures are in effect for entering the empty water tank. Be sure the confined space blower is turned on supplying fresh air into the bottom of the tank.

4 Procedure

1. Start cooling the glycol and bubble chamber to between 1 C and 5 C using the chiller and cooling coils. This may take several days.

2. Place the CF3I storage container on a scale capable of 0.5 kg resolution. Wrap a heat blanket on the container or have some alternate means of adding heat. Record the starting net weight/mass.

3. Place a clean, high purity, 10 gallon (37.9 liter) Alloy Products Corporation in an open fluid bath on a scale capable of 0.5 kg resolution. This will be the intermediate distillation vessel. Connect a chiller to the fluid bath. Record the starting net weight/mass.

4. Connect a clean Teflon fill transfer line between the CF3I storage container and a valve on the gas port of the intermediate vessel. A fine particulate filter should be part of the transfer line. The transfer line also will need a tee to a vacuum pump for line evacuation.

5. Pull a vacuum on the transfer line and intermediate vessel. Confirm that the transfer line and vessel is clean and leak free by achieving an ultimate pressure equal to the pump ultimate pressure and a rate of rise of less than 100 microns per hour. Backfill with CF3I gas. Repeat an evacuation and backfill with CF3I at least 2 more times. The saturated pressure of the CF3I in the storage container at room temperature is 62.6 psia = 47.9 psig. This will be the gas pressure in the transfer line and vessel. Leave the valve on the supply cylinder and the intermediate vessel open.

6. Record the masses/weights on the scales. Start cooling the intermediate distillation vessel to 1 C (34 F).

7. CF3I should be condensing into the intermediate vessel. Periodically record the time and masses/weights to determine the transfer rate. See next step if transfer slows or stops. Apply heat if necessary to the supply cylinder to keep it at room temperature or slightly above. Maximum temperature allowed is 37 C (100 F).

8. If CF3I condensation halts then suspect some non-condensable gas blocking the transfer line. The non-condensable gas will need to be removed in order to continue. Close the valve on the intermediate container and close the gas supply cylinder valve. A tee with pumping line to a dry vacuum pump is part of the transfer line. Evacuate the non-condensable gas. Crack open bubble chamber valve briefly (do not allow vacuum pressure to go above atmospheric pressure) to remove any non-condensable gas in the bubble chamber volume. When the vacuum is near the ultimate achievable, isolate the vacuum pump. Backfill the transfer line with CF3I from the supply cylinder. Repeat a second time if desired. Resume operation by opening the valves.

9. Close the valves when 29.0 kg (63.9 lbs) has been transferred.

10. Change the fluid bath temperature for the intermediate vessel to 20 C (68 F).

11. Connect the Teflon fill transfer line between the intermediate distillation vessel and the downstream purge port of the Carten valve, MV-81 on bubble chamber.

12. Pull a vacuum on the transfer line. Confirm that the transfer line is clean and leak free by achieving an ultimate pressure equal to the pump ultimate pressure and a rate of rise of less than 100 microns per hour. Backfill with CF3I gas. Repeat an evacuation and backfill with CF3I at least 3 more times. The saturated pressure of the CF3I in the storage container at room temperature is 62.6 psia = 47.9 psig.

This will be the gas pressure in the transfer line. Leave the valve on the supply bottle open.

13. Connect a valve and ¼" tube to CF3I bubble chamber side purge port, MV-83. This will be used to extract the water from the bubble chamber. Place the end of the tube into a carboy capable of holding 4 liters of water. Know the tare weight or exact volume of the carboy. The mass or volume of water extracted will need to be determined.

14. The system should still be in the "compressed" state. At 200 psig, the pump position set point should be 1.6". Adjust the pressure in the hydraulic system to 35 to 40 psig (49.5 psia to 54.5 psia) by adjusting PRV-10 to pressurize the air tank to roughly 10 psig on PIT-7. Set the pump position set point at ZT-6 = 0.2". When the set point, ZT-6 = 0.2" is achieved, stop the pump.

15. Turn on the video cameras and LED lighting grid to enable "live" viewing of the quartz jar. Confirm that the air cooling blower for the camera enclosure is on. A little pink streamer should be blowing indicating exhaust air flow.

16. Open valve MV-83 to purge out water until the bellows position, ZT4 decreases from +0.625" to -1.375". Close MV-83. The change in bubble chamber volume is 2.0 liters per inch of travel, ZT4. Therefore the volume of water removed should be 4 liters. The glycol system volume change is 0.20 liters per inch of glycol piston travel. So the glycol piston, ZT6 will need to move 8" for this much volume change.

17. Record the starting mass/weight of the intermediate vessel in the fluid bath.

18. Open the transfer line/Carten downstream purge port valve, MV-81 to allow CF3I to flow into the bubble chamber. The CF3I pressure (48 psig) is greater than the glycol pressure (40 psig) so the bubble chamber will expand. The glycol piston ZT6 will travel to 0.0". When the piston bottoms out at 0.0", the glycol pressure will equalize to the CF3I pressure inside the bubble chamber, 48 psig. The stop plate will move up as gas flows into the bubble chamber. The ZT4 position will increase. When ZT-4 reaches approximately +0.5", close or throttle the main Carten valve, MV-81 or the valve at the intermediate vessel to try to keep ZT-4 in the range of 0.0" to 1.0".

19. CF3I should be condensing into the bubble chamber and collecting under the water at the bottom of the quartz jar. If the CF3I appears to be in the form of snow or ice, then increase the glycol temperature. (See what-if) The saturation pressure of the CF3I at 1 C is 34.1 psia = 19.5 psig. The quartz jar has an inside diameter of 29.2 cm. The CF3I level will be 5.0 cm up from the bottom when 1 liter (2 kg) of CF3I accumulates. The CF3I level will be 6.2 cm up from the bottom when 1.5 liters (3.0 kg) of CF3I accumulates.

20. If CF3I condensation halts, suspect some non-condensable gas blocking the transfer line. The non-condensable gas will need to be removed in order to continue. Close MV-81 (isolating the bubble chamber) and close the intermediate vessel valve. Evacuate the non-condensable gas. Isolate the vacuum pump. Backfill the transfer line with CF3I from the supply cylinder. Repeat a second time if desired. Crack open the water withdrawal valve, MV-83 to remove any non-condensable gas in the bubble chamber volume. Close MV-83 when water starts to be withdrawn. Resume operation.

21. When nearly 4 liters of CF3I accumulates, the valve on the transfer line (either MV-81 or valve at supply cylinder) will need to be closed completely to keep ZT-4 from exceeding the desired range. When this occurs, proceed to the next step.

22. Close the valve, MV-81. The glycol pressure may reduce from 48 psig to 40 psig as any remaining gas in the bubble chamber condenses and the glycol piston will come off of its 0.0" ZT6 position.

23. Open valve MV-83 to purge out water until the bellows position, ZT4 decreases by about 2" to -1. 5". Close MV-83. The change in bubble chamber volume is 2.0 liters per inch of travel, ZT4. Therefore the volume of water removed should be 4 liters. The glycol system volume change is 0.20 liters per inch of glycol piston travel. So the glycol piston, ZT6 will need to move 8" for this much volume change.

24. Repeat steps 7, 8, 9, and 10 until 13.2 liters (13.2 kg) of water has been removed from the system and 29 kg of CF3I has been transferred into the bubble chamber. Try to adjust system volumes (while trying to remove the right amount of water) so that after the filling is completed, ZT-4 position is -0.92" (assuming the system temperature is 1 Celsius).

25. Close the valve, MV-81.

26. Close the valve on the intermediate vessel.

27. Vent the transfer line via the downstream purge port of the Carten valve. Do not vent the CF3I directly at personnel or breathe it. It is an irritant.

28. Remove the transfer line and other equipment.

29. Increase the air tank pressure from about 10 psig to 55 psig to give a glycol pressure of approximately 200 psig.

30. Change the pump set point to 2.0". (SP is somewhat arbitrary) Activate the pump in automatic by pushing the "start" pump button on the CMore screen.

<u>Warming to operating temperature</u>

1. Change the temperature set point for the chiller to 20 C.

2. Confirm that the glycol pump is in automatic so that it will move glycol into the diaphragm tank as the bubble chamber volume expands and displaces glycol.

3. The liquid volume of the CF3I will expand from 13.15 to 13.73 liters during the 1 C to 20 C temperature change. The stop plate will move from -0.92" to -0.20".

<u>Operating at 20 C</u>

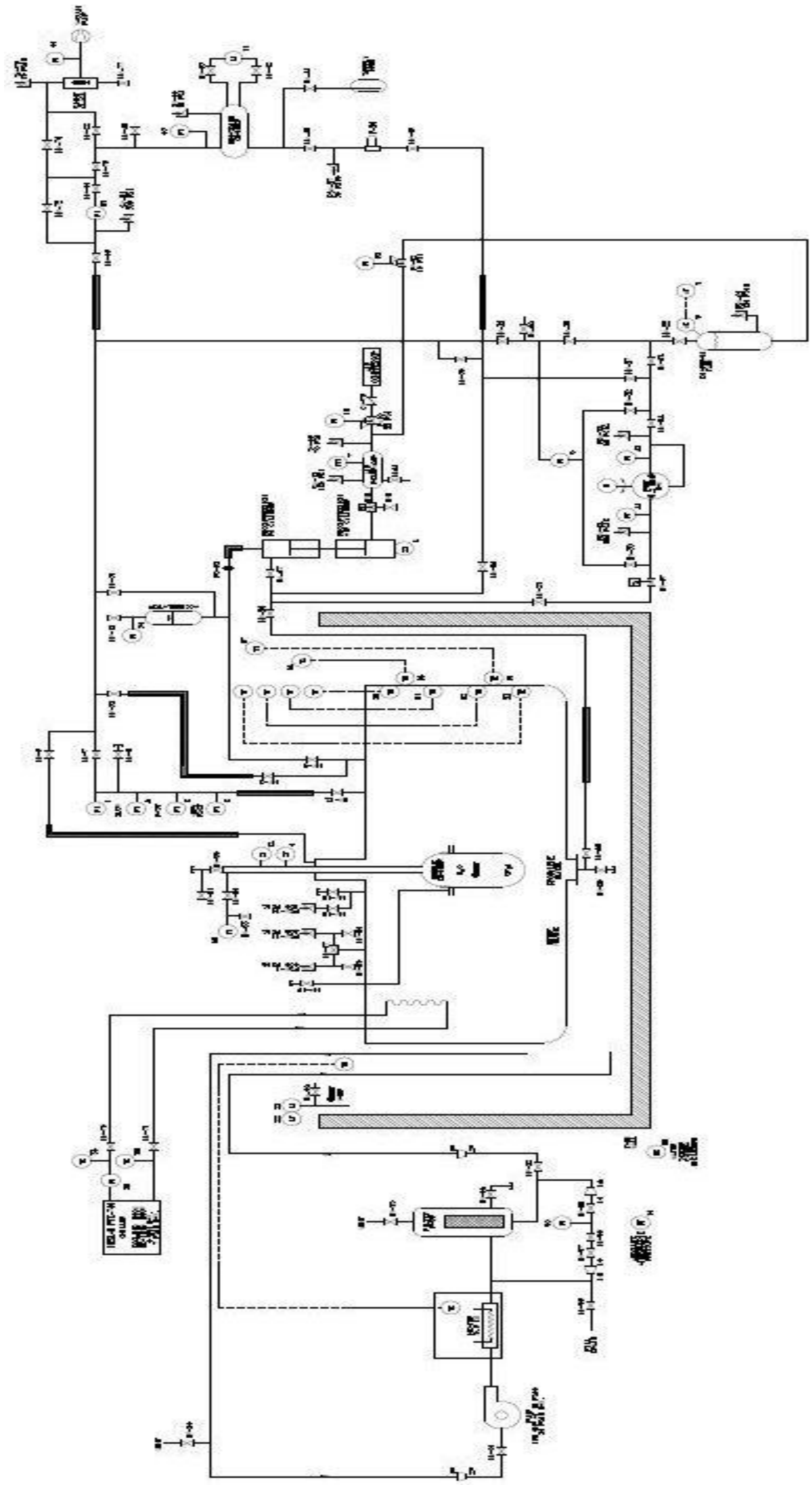
1. The volume change of the 29 kg of CF3I from the 200 psig compressed state to 5 psig superheated state is 0.12 liters at 20 C. This is a stop plate motion 0.06" and a glycol piston motion of 0.5". Total glycol piston travel will likely be about 2.0". This is a summation of the 0.5" to compress the CF3I and about 1.5" to compress dissolved air in the glycol system.

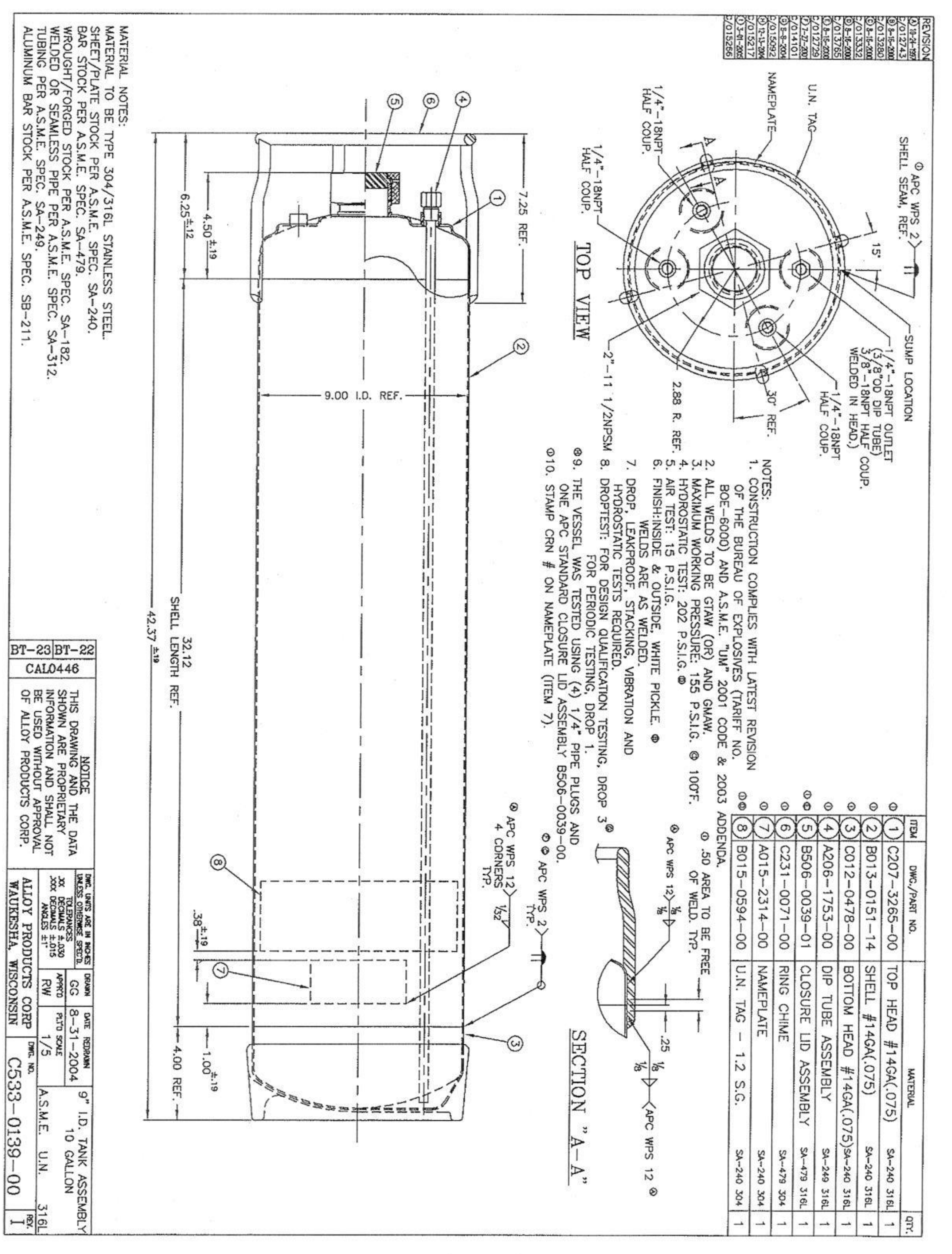
2. The bubble chamber is ready for automatic or manual de-compression and fast re-compression cycles.

<u>Operating at 40 C</u>

1. The volume change of the 29 kg of CF3I from the 200 psig compressed state to 5 psig superheated state is 0.12 liters at 40 C. This is a stop plate motion 0.09" and a glycol piston motion of 0.65". Total glycol piston travel will likely be about 2.1". This is a summation of the 0.65" to compress the CF3I and about 1.5" to compress dissolved air in the glycol system.

2. The bubble chamber is ready for automatic or manual de-compression and fast re-compression cycles.





Alloy Products Corporation 10 gallon intermediate vessel.

What – If Analysis

1. What if something unexpected happens, can we safely stop the transfer? We can stop during the pump and backfill steps. We can stop during the expelling of

water with no adverse problems. We can stop the CF3I transfer at any point. The gas that remains to be liquefied will eventually liquefy and the expansion chamber will compress to a position within the working range.

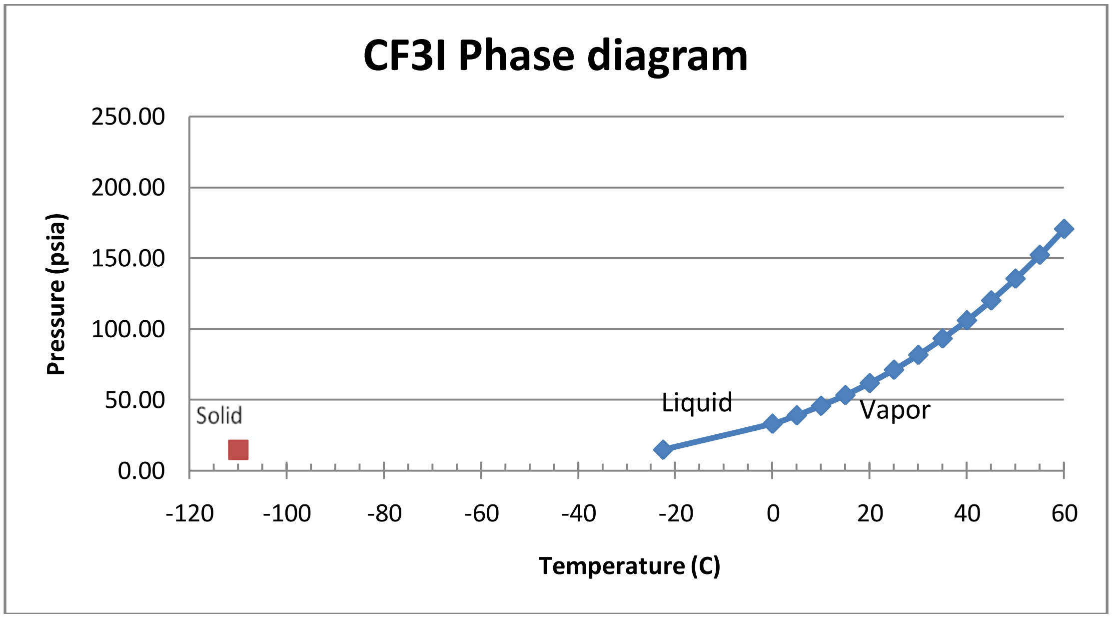
2. What if we accidently let the stop plate bottom out when we are withdrawing water? If the stop plate bottoms out, then an external differential pressure of 40 psid will develop across the expansion chamber and jar. That would exceed the bellows design pressure rating of 33 psid. Hyspan, the bellows manufacturer states that no permanent deformation will occur for up to 1.5 times the design pressure rating or 49.5 psid. Therefore while bad, no permanent damage should occur to the bellows. The quartz jar has a compressive strength exceeding 190 psid, so no damage will occur to the quartz jar.

3. What if we accidently let the stop plate hit the shoulder bolts at ZT-4 = +1.125"? This could occur during the CF3I transfer. An internal differential pressure of about 8 psid could develop across the bubble chamber wall. This differential is well within the working pressure design of the bubble chamber and no problem is forseen.

4. What if CF3I condensation halts and we suspect we have some non-condensable gas blocking the transfer line? The non-condensable gas will need to be removed in order to continue. We Close MV-81 (isolating the bubble chamber) and close the gas supply cylinder valve. A tee with pumping line to a dry vacuum pump will need to be part of the transfer line. Evacuate the non-condensable gas. Isolate the vacuum pump. Backfill the transfer line with CF3I from the supply cylinder. Crack open the water withdrawal valve, MV-83 to remove any non-condensable gas in the bubble chamber volume. Close MV-83 when water starts to be withdrawn. Resume operation.

5. What if we see snow or ice CF3I, do we just warm up the glycol. Do we stop? We cannot change the pressure of the transfer because the CF3I supply cylinder is at saturated conditions at room temperature. So the only thing we can do is warm the temperature of the glycol. We do not need to stop. As a suggested starting point, raise the chiller temperature to 5 C.

6. What if the temperature of the glycol gets too cold and the water freezes? If the water starts to freeze, then the CF3I will no longer be able to condense. The water could expand causing physical damage to the bellows. The chiller set point should never be lower than 1 C. The heat load from the coolant piping and vessel surfaces will guarantee that the water will be greater than the chiller set point and therefore will not freeze.



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